The observation of slip bands in molybdenum single crystals by X-ray diffraction*

The etch pit technique is a very simple method for the direct observation of dislocations and slip bands in single crystals. However, its use is often limited to certain crystallographic planes. For example, at present there is no report in the literature of an etchant which will reveal dislocations on {112} planes of molybdenum. The Berg-Barrett X-ray micrography technique can be used to observe slip bands on the {112} planes. However, before using this method in studies of the growth of slip bands in molybdenum, it was deemed necessary to establish a correspondence between the X-ray contrast and the total slip band length (length of the trace containing dislocations associated with the slip band). To accomplish this goal X-ray topographs of slip bands were compared with the etch pit pattern produced by an electrolytic etchant developed by G. C. Das[1]. These observations were made on a {124} surface of molybdenum (since the etchant does not reveal dislocation on a {112} surface).

A {124} surface of molybdenum was prepared by spark erosion followed by electrolapping with a 1 molar $FeCl_2$ solution. The surface was electropolished in H_2SO_4 and C_2H_5OH (1:6) and electrolytically etched in H_2SO_4, CH_3OH, and $HClO_3$ (1:1:1). Then fresh slip bands were introduced in the crystals by indenting with a needle[2]. The Berg-Barrett

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*This work was supported by the U. S. Atomic Energy Commission.

X-ray exposures were taken. The crystal surface was electropolished to clean it and the fresh dislocations were electrolytically etched.

To interpret the X-ray pictures properly, two things must be realized. First, the X-ray geometry produces a distorted picture of a crystal surface[3]. Second, the slip bands will show up as darkened areas. Figs. 1 and 2 show that the darkened area corresponds in size and shape to the etch pitted area: this indicates that the X-ray technique reveals the true extent of the slip band. Figs. 3 and 4 illustrate an advantage of the X-ray technique: the background dislocations do not appear on the X-ray topograph and therefore the leading edge of the slip band is more readily determined from the X-ray topograph than from the etched surface. It is concluded that the length of slip bands measured on X-ray topographs are sufficiently accurate to be used for the study of slip band growth.

The authors thank Professor D. F. Stein for supplying the crystals. R. F. Boyce wishes to express his thanks to NASA for fellowship support during the course of this work.

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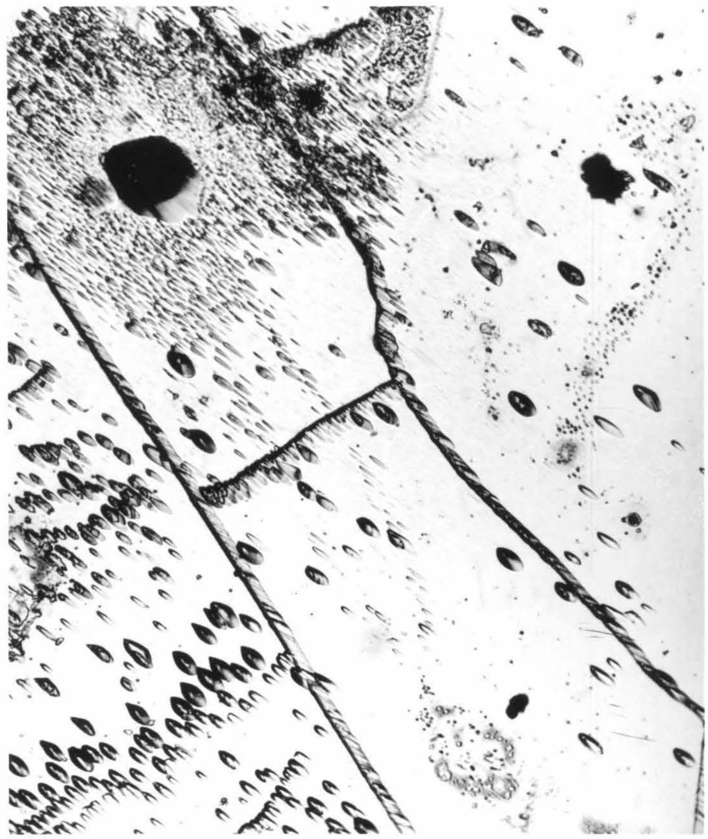
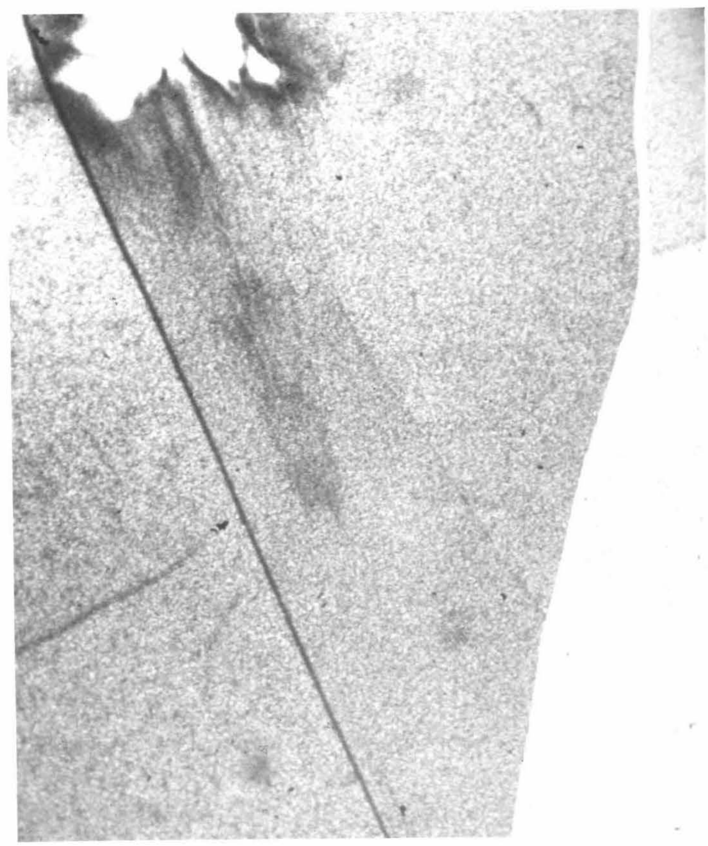
FIGURE 1



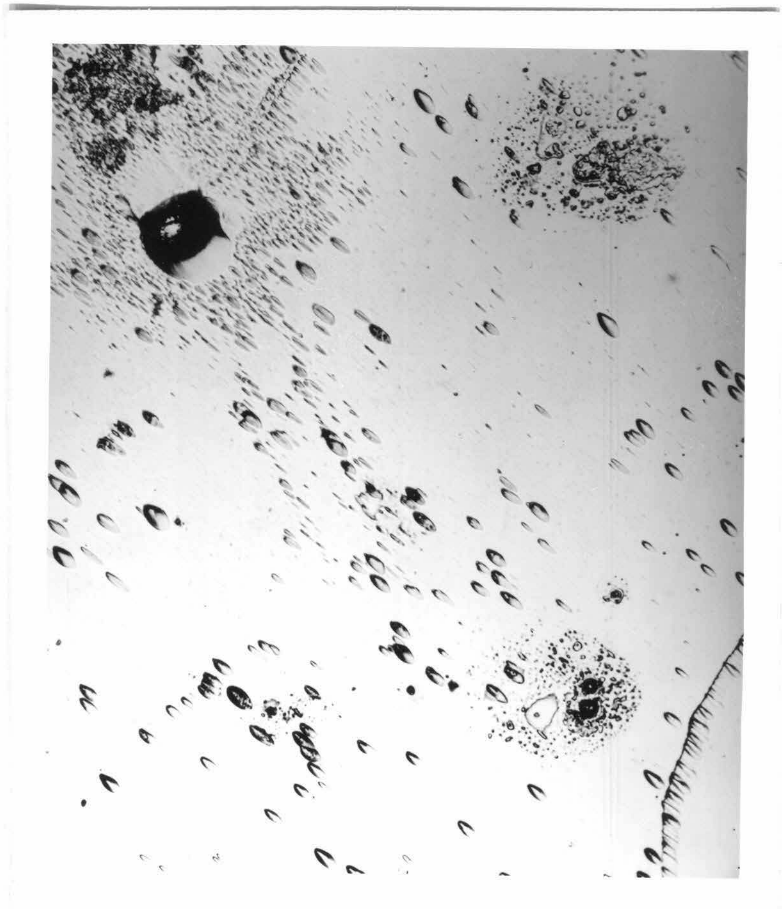
Slip band A $\{124\}$ surface of Mo electrolytically etched, X 236.7.

FIGURE 2



Slip band A Berg-Barrett X-ray micrograph (321) reflecting plane 3° from zero layer, $CuK\alpha$, X 362.8.

FIGURE 3



Slip band B {124} surface of Mo electrolytically etched, X 236.7.

FIGURE 4

Slip band B Berg-Barrett X-ray micrograph (321) reflecting plane 3^{o} from zero layer, CuKα, X 362.8.